
सौन्दर्य-प्रसाधन उद्योग के लिये
मिथाइल-पी-हाइड्रोक्सीबेंजोएट /
मिथाइल पैराबीन — विशिष्ट
(तीसरा पुनरीक्षण)

**Methyl-*p*-Hydroxybenzoate /
Methyl Paraben for Cosmetic
Industry — Specification**
(*Third Revision*)

ICS 71.100.70

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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was originally published in 1968 and subsequently revised in 1977 and 1985. Initially, in the first revision, the Cosmetics Sectional Committee had decided to indicate the essential and the optional requirements separately. Subsequently, this was found non-implementable for certification and therefore, in second revision, only a single set of requirements was stipulated. In second revision, method of 'Assay' was revised and test for sulphate was modified to align the test methods with Indian Pharmacopoeia (I.P.) 1966. The requirement of pH was deleted and replaced with the test reaction in line with I.P. Due to strong reservations regarding the use of potassium cyanide in a cosmetic testing laboratory, potassium cyanide method for test of heavy metals was replaced by hydrogen sulphide method.

In this revision, the 'Assay' limit has been revised to align with the I.P. 2014. The volumetric method of 'Assay' has also been replaced by HPLC method, in which retention times of various parabens are different, thus, these can be individually identified and quantified.

Methyl-*p*-hydroxybenzoate is used as preservative in the cosmetic industry; it prevents the growth of micro-organisms.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHYL-*p*-HYDROXYBENZOATE / METHYL PARABEN FOR COSMETIC INDUSTRY — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for methyl-*p*-hydroxybenzoate / methylparaben for cosmetic industry.

2 REFERENCES

The following standard contain provisions, which through reference in this text, constitutes provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

<i>IS No.</i>	<i>Title</i>
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a white, non-hygroscopic powder. The material is slightly soluble in water (1 part in 400 parts) and freely soluble in ethyl alcohol.

3.2 The material shall also comply with the requirements given in Table 1 when tested as prescribed in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The commonly used size and material of construction of containers is 5 kg gusseted polythene bag which is further packed in 5-ply corrugated box of grammage 5×100 gsm or shall be subject to agreement between the purchaser and the supplier.

4.2 Marking

4.2.1 The container shall be legibly marked with the following information:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Net content;
- d) Month and year of manufacture (MM/YY);
- e) Use before..... (Month and year MM/YY, or months/years from the date of manufacture) to be declared by the manufacturer;
- f) Batch number; and
- g) Any other information required by statutory authorities.

**Table 1 Requirements for Methyl-*p*-hydroxybenzoate/
Methylparaben for Cosmetic Industry**

(Clause 3.2)

SI No.	Characteristics	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Melting range, °C	125 to 128	A
ii)	Arsenic (as As ₂ O ₃), ppm, <i>Max</i>	2	B
iii)	Heavy metals (as Pb), ppm, <i>Max</i>	20	C
iv)	Assay, <i>Min</i>	98.0	D
v)	Sulphated ash, percent by mass, <i>Max</i>	0.1	E
vi)	Chlorides	To pass the test	F
vii)	Sulphates	To pass the test	G
viii)	Reaction	To pass the test	H

4.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

The method for preparing representative test samples of the material and criteria for conformity shall be as prescribed in **Annex J**.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water [*see IS 1070 : 1992 'Reagent grade water (third revision)'*] shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

ANNEX A

[Clause 3.2, Table 1, SI No. (i)]

DETERMINATION OF MELTING RANGE

A-1 APPARATUS

A-1.1 Oil Bath Consisting of liquid paraffin in a glass container.

A-1.2 Heat Source Electrical or open flame, capable of being controlled at a desired rate of heating.

A-1.3 Capillary Tubes Of glass, 10 cm long and 0.8 to 1.2 mm in internal diameter.

A-1.4 Thermometer Having least count 1°C.

A-2 PROCEDURE

A-2.1 Reduce the sample to a fine powder. Fill in a capillary tube, which is sealed at one end, sufficient quantity of dry powder to form a column 2.5 to 3.6 mm in height when packed down as closely as possible.

A-2.2 Heat the bath until a temperature approximately 30°C below the expected melting point is reached.

Attach capillary tube containing the powdered sample to the thermometer by wetting both with a drop of liquid paraffin and adjust its height so that the material in the capillary is in level with the thermometer bulb. Return the thermometer to the bath and continue heating with constant stirring at a rate of approximately 3°C per min until a temperature 3° below the expected melting point is attained. Then carefully regulate the rate to 1°C per min until the sample is completely melted.

A-2.3 The temperature at which the column sample is observed to collapse definitely against the side of the tube at any point shall be taken as the beginning of the melting and the temperature at which the sample becomes liquid throughout shall be taken as the end of melting.

A-2.4 Both the temperatures noted shall fall within the specified melting range.

ANNEX B

[Clause 3.2, Table 1, SI No. (ii)]

DETERMINATION OF ARSENIC

B-1 OUTLINE OF THE METHOD

Arsenic present in a solution of the material is reduced to arsine, which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

B-2 REAGENTS

B-2.1 Concentrated Sulphuric Acid [see IS 266 : 1993 'Sulphuric acid — Specification (*third revision*)']

B-2.2 Concentrated Nitric Acid [see IS 264 : 2005 'Nitric acid — Specification (*third revision*)']

B-3 PREPARATION OF SAMPLE

Weigh 1.000 g of the sample in a Kjeldahl flask of 500 ml capacity. Add 5 to 10 ml of concentrated nitric acid followed by 4 ml of concentrated sulphuric acid. Heat cautiously. Add drop by drop more nitric acid, if required, from a pipette to speed up the oxidation of the sample. The total amount of nitric acid shall be noted for use in the control test. When oxidation is complete, the solution is a clear and faint yellow; at that stage, add 20 ml of water and again boil to fuming. Ensure removal of all nitric acid.

B-4 PROCEDURE

Carry out the test for arsenic with the solution prepared in **B-3** as prescribed in IS 2088. For comparison, prepare a stain using 0.002 g of arsenic trioxide.

ANNEX C

[Clause 3.2, Table 1, SI No. (iii)]

TEST FOR HEAVY METALS

C-1 OUTLINE OF THE METHOD

The color produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.

C-2 APPARATUS

C-2.1 Nessler Cylinders — 50-ml capacity.

C-3 REAGENTS

C-3.1 Concentrated Hydrochloric Acid [*see* IS 265 : 1993 'Hydrochloric acid — Specification (*fourth revision*)']

C-3.2 Concentrated Nitric Acid (*see* IS 264)

C-3.3 Dilute Acetic Acid — 6 percent acetic acid (60 ml diluted to 1000 ml water).

C-3.4 Hydrogen Sulphide Solution — Saturated.

C-3.5 Standard Lead Solution — Dissolve 1.600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water.

One milliliter of this solution contain 0.01 mg of lead (as Pb).

C-4 PROCEDURE

C-4.1 Ignite 1.000 g of the material in a silica dish until completely ashed. To the residue add 1 ml of hydrochloric acid and 0.5 ml nitric acid and evaporate to dryness on the steam bath. Dissolve the residue in 5 ml hot water and 1 ml dilute acetic acid and transfer to Nessler cylinder washing in with water. In the second Nessler cylinder, place 1 ml of dilute acetic acid and add 2 ml of standard lead solution.

C-4.2 Add 10 ml of hydrogen sulphide solution to each Nessler cylinder and make up the volume with water to 50 ml. Mix and allow to stand for 10 min. Compare the colour produced in the two Nessler's cylinders. Blank determination without samples are recommended to avoid errors arising out of reagents.

C-5 RESULTS

The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the second Nessler cylinder.

ANNEX D

[Clause 3.2, Table 1, SI No. (iv)]

ASSAY

D-1 REFERENCE STANDARD SOLUTION

Dissolve 50 mg of methyl-*p*-hydroxybenzoate reference standard in 2.5 ml of methanol and dilute to 50 ml with the mobile phase. Dilute 10 ml of this solution to 100 ml with the mobile phase.

D-2 SAMPLE SOLUTION

Dissolve 50 mg of the sample in 2.5 ml of methanol and dilute to 50 ml with the mobile phase. Dilute 10 ml of this solution to 100 ml with the mobile phase.

D-3 CHROMATOGRAPHIC SYSTEM

D-3.1 Stainless Steel Column — 15 cm × 4.6 mm, packed with octadecylsilane bonded to porous silica (5 µm).

D-3.2 Mobile Phase — A mixture of 35 volumes of 0.68 percent solution of potassium dihydrogen phosphate (*w/v*) and 65 volumes of methanol.

D-3.3 Flow Rate — 1.3 ml/min.

D-3.4 Spectrophotometer Set — 272 nm.

D-3.5 Injection Volume — 10 µl.

D-4 PROCEDURE

Inject reference standard solution and sample solution to the instrument and record the area of reference standard and sample.

D-5 CALCULATION

$$\text{Assay, percent} = \frac{\text{Area of sample} \times \text{Weight of reference standard} \times \text{Purity of reference standard}}{\text{Area of reference standard} \times \text{Weight of sample}}$$

ANNEX E

[Clause 3.2, Table 1, SI No. (v)]

DETERMINATION OF SULPHATED ASH

E-1 REAGENT

E-1.1 Dilute Sulphuric Acid — Approximately 4 N.

E-2 PROCEDURE

Weigh accurately 1 to 2 g of sample in a tared crucible. Ignite until thoroughly charred, cool, and moisten with 1 ml of dilute sulphuric acid and cautiously ignite until the carbon is completely consumed. Conduct the ignition at a place protected from air currents and use as low a temperature as possible to effect the combustion

of carbon. When carbon has completely disappeared, cool the crucible in a desiccator and weigh.

E-3 CALCULATION

$$\text{Sulphated ash, percent by mass} = \frac{m \times 100}{M}$$

where,

m = mass, in g, of the residue obtained, and

M = mass, in g, of the sample taken for the test.

ANNEX F

[Clause 3.2, Table 1, SI No. (vi)]

TEST FOR CHLORIDES

F-1 APPARATUS

F-1.1 Nessler Cylinders — 50 ml capacity.

F-2 REAGENTS

F-2.1 Silver Nitrate Solution — 2 percent.

F-2.2 Dilute Nitric Acid — 4 N.

F-2.3 Standard Hydrochloric Acid — Exactly 0.01 N.

F-3 PROCEDURE

F-3.1 Heat 2.000 g of the sample with 100 ml of water cool, and water to restore to the original

volume, and filter through cotton. Preserve the filtrate for test for chlorides and sulphates (Annex G). Take 50 ml of the filtrate in a Nessler cylinder and add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Carry out a control test in another Nessler cylinder using 1.0 ml of standard hydrochloric acid and the same quantities of other reagents as used with the sample. Allow the cylinders to stand for 5 min protected from direct sunlight and compare the turbidity.

F-3.2 The sample shall be taken to have passed the test if the turbidity produced with the sample is not greater than that in the control test.

ANNEX G

[Clause 3.2, Table 1, SI No. (vii)]

TEST FOR SULPHATES

G-1 REAGENTS

G-1.1 Dilute Hydrochloric Acid — 4 N.

G-1.2 Barium Chloride Solution — 10 percent.

G-2 PROCEDURE

G-2.1 Take a 10 ml aliquot of the filtrate preserved in F-3.1 in a Nessler cylinder, add a few drops of dilute hydrochloric acid and a few drops of barium chloride solution. Mix and allow to stand for 10 min.

G-2.2 The sample shall be taken to have passed the test, if no turbidity is produced within 10 min.

ANNEX H

[Clause 3.2, Table 1, SI No. (viii)]

REACTION

H-1 PROCEDURE

Heat 0.5 g of sample in 10 ml of water to 80°C, cool and filter. The sample shall be taken to have passed the

test if the filtrate is neutral or slightly acidic to solution of litmus.

ANNEX J

(Clause 5)

**SAMPLING OF METHYL-*p*-HYDROXYBENZOATE /
METHYLPARABEN FOR COSMETIC INDUSTRY**

J-1 GENERAL REQUIREMENTS OF SAMPLING

In drawing, preparing storing and handling test samples, the following precautions and directions shall be observed.

J-1.1 The samples shall not be taken in an exposed place.

J-1.2 The sampling instrument shall be clean and dry.

J-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

J-1.4 To draw a representative sample, the contents of each container selected for sampling and shall be mixed as thoroughly as possible by suitable means.

J-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

J-1.6 The samples containers shall be of such size that they are almost completely filled by the sample.

J-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

J-2 SCALE OF SAMPLING**J-2.1 Lot**

All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture,

the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

J-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the Requirements of this specification.

J-2.2 The number of container (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be as given in Table 2.

J-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, random number tables shall be used [see IS 4905 : 2015 Random sampling and randomization procedures (*first revision*)]. In case such tables are not available, the following procedure may be adopted.

**Table 2 Number of Containers to be
Selected for Sampling**

(Clause J-2.2)

<i>Lot Size</i>	<i>Number of Containers to be Selected</i>
(N)	(n)
(1)	(2)
Up to 3	Each container
4 to 50	3
51 to 150	4
151 to 300	5
301 to 500	7
501 and above	10

Starting from any container, count them as 1, 2, 3,, up to r and so on in a systematic manner, where r is the integral part of N/n . Every r^{th} container thus counted shall be withdrawn from the lot.

J-3 TEST SAMPLE AND REFEREE SAMPLE**J-3.1 Preparation of Test Samples**

J-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in Table 1 and shall not be less than 250 g.

J-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 0.5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

J-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **J-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test samples each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used for-the referee.

J-3.2 Referee Sample

The referee samples shall consist of a composite sample (**J-3.1.2**) and a set of individual samples (**J-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

J-4 NUMBER OF TESTS

J-4.1 Test for assay shall be conducted on each of the individual samples.

J-4.2 Tests for the remaining characteristics given in Table 1 shall be conducted on the composite sample (**J-3.1.2**).

J-5 CRITERIA FOR CONFORMITY**J-5.1 For Individual Samples**

The mean (\bar{x}) and range (R) for the test results shall be calculated (range being the difference between the maximum and the minimum test results).

J-5.1.1 The lot shall be declared to have satisfied the requirements for assay if the value of the expression $(\bar{x} - 0.6R)$ is equal to or greater than 98.0.

J-5.2 For Composite Sample

The test results on the composite sample shall meet the corresponding requirements specified in **3** and Table 1.

J-5.3 A lot shall be declared as conforming to this specification if it satisfies the requirements for each of the characteristics listed in Table 1. If the requirements for any of the characteristics are not met the lot shall be declared to have not satisfied the requirement of the specification.

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This Indian Standard has been developed from Doc No.: PCD 19 (12559).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi